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IS 12107-2 (1987): Methods of chemical analysis of alumino-silicate refractory materials, Part 2: Determination of silica [MTD 13: Ores and Raw Materials]

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ALUMINO SILICATE REFRACTORY MATERIALS

PART 2 DETERMINATION OF SILICA

Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee, SMDC 35 [Ref : Doc : SMDC 35 (3002)]

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1. Scope — This standard (Part 2) covers a method for determination of silica in alumino silicate refractory materials.

2. Determination of Silica by Gravimetric Method

2.1 *Outline of the Method* — The sample is decomposed by fusion mixture and extracted with acids. Silica is determined by dehydration and baking followed by hydrofluorization. The residual silica is determined photometrically and combined.

2.2 *Reagents*

2.2.1 *Fusion mixture* — Mix equal amount of sodium carbonate and potassium carbonate.

2.2.2 *Potassium iodide* — 1 percent (*m*/*v*).

2.2.3 *Dilute hydrochloric acid* — 1 : 1, 1 : 3 and 1 : 20 (*v*/*v*).

2.2.4 *Dilute sulphuric acid* — 1 : 1 (*v*/*v*).

2.2.5 *Methyl alcohol*

2.2.6 *Concentrated hydrochloric acid* (*r.d.* = 1·16) — Conforming to IS : 265-1976 'Specification for hydrochloric acid (*second revision*)'.

2.2.7 *Hydrofluoric acid* — 40 percent.

2.2.8 *Potassium bisulphate* — Solid.

2.2.9 *EDTA solution* (*0·05 M*) — Dissolve 18·611 g of EDTA (disodium salt) in 1 litre of water.

2.2.10 *Phenolphthalein indicator solution* — 0·1 percent (*m*/*v*) in ethanol.

2.2.11 *Dilute ammonia solution* — 1 : 1 (*v*/*v*).

2.2.12 *Ammonium molybdate solution* — Dissolve 10 g of ammonium molybdate [$(NH_4)_6 MO_7O_{24}.4H_2O$] in 100 ml of water, filter if necessary.

2.2.13 *Oxalic acid solution* — 10 percent (*m*/*v*).

2.2.14 *Ascorbic acid solution* — 10 percent (*m*/*v*) stored in an amber coloured bottle (stable up to 5 days).

2.2.15 *Standard silica solution*

a) Fuse 0·20 g of pure silica with 5·0 g of fusion mixture (*see* **2.2.1**) in a platinum crucible. Cool and dissolve the melt in 300 ml of water containing 20 g of sodium hydroxide in a polythene beaker. Warm the solution on a water bath and dilute to 500 ml in a volumetric flask. Store in a polythene bottle.

b) Prepare immediately before use, a standard working solution containing 0·04 mg/ml of silica (SiO_2) by diluting the above stock solution.

2.3 *Procedure*

2.3.1 *Decomposition of the sample* — Weigh 1·000 g of finely ground test sample [*see* **2.3** of IS : 12107 (Part 1)-1987 Methods of chemical analysis of alumino silicate refractory materials: Part 1 Determination of loss on ignition] into a platinum crucible. Add 6 g of fusion mixture and mix with a platinum rod, add 2-3 drops of potassium iodide solution and mix again. Heat the contents over a gas burner cautiously. Increase the temperature and keep it for 20 minutes with

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occasional swirling of the melt with a platinum tipped tong. Cool and observe that a transparent glass is obtained (this is an indication for quantitative decomposition of the sample).

2.3.1.1 Place the crucible into a porcelain dish containing 50 ml of dilute hydrochloric acid (1 : 1) and 2 ml of dilute sulphuric acid and ensure the mass is completely detached from the crucible. Cover the dish with a watch glass when the reaction ceases, wash the crucible with water.

2.3.1.2 Add 10 ml of methyl alcohol and place the dish on a steam bath to remove boron trioxide by treating 2-3 times with 10 ml of methyl alcohol and evaporate the solution to dryness until the smell of hydrochloric acid is no longer detected. Finally, place the dish in an air oven and bake it at 110°C. Cool the dish and add 10 ml of concentrated hydrochloric acid and 40 ml of hot water and stir.

2.3.2 *Determination of major silica by gravimetric method* — Cover the dish and allow the mixture to digest on a steam bath for 10 minutes. Filter through Whatman No. 42 filter paper. Transfer the silica to filter paper with hot dilute hydrochloric acid (1 : 20), scrubbing the basin with a rubber-tipped glass rod. Wash the precipitate five times with hot dilute hydrochloric acid (1 : 20) and then with hot water until free from chlorides. Reserve the filterate (solution *A*).

2.3.2.1 Transfer the paper and the precipitate to an ignited and weighed platinum crucible. Ignite at a low temperature until the precipitate is free from carbonaceous matter and then heat in the furnace at 950-1 000°C to constant mass. Moisten the contents with water, add 5 drops of dilute sulphuric acid and 10 ml of hydrofluoric acid. Evaporate to dryness on sand bath or hot plate.

2.3.2.2 Heat the crucible and residue cautiously first, over gas flame and finally for 2 minutes at 950-1 000°C in the furnace, cool in a desiccator and weigh to constant mass. If the residue weighs more than 10 mg, repeat the treatment with dilute sulphuric acid and hydrofluoric acid.

2.3.3 *Preparation of main solution* — Fuse the residue after hydrofluoric acid treatment with 1 g of potassium bisulphate, dissolve the melt in water containing a few drops of concentrated hydrochloric acid and add the solution to the main filterate (solution *A*) (*see* **2.3.2**). Cool and dilute to 500 ml in a volumetric flask.

2.3.4 *Determination of residual silica by photometric method* — Transfer 10 ml of main solution (*see* **2.3.3**) to a 100-ml volumetric flask. Add 5 ml of EDTA solution and a drop of phenolphthalein solution and then dilute ammonia solution until the colour charges to red. Add 5 ml of ammonium molybdate solution and allow to stand for 10 minutes. Add 10 ml of oxalic acid followed by 5 ml of ascorbic acid and dilute to 100 ml. Allow the solution to stand for half-an-hour for development of full colour. Measure the absorbance at 810 nm against a reagent blank solution prepared in the same manner without the sample at 25 ± 5°C. Compute the silica content from the calibration curve.

2.3.4.1 *Calibration curve* — Establish a calibration curve by taking 0, 1, 2, 3 and 5 ml of standard silica solution (1 ml = 0·04 mgSiO_2) into five 100-ml volumetric flasks. Carry out the entire procedure as given in **2.3.4** without the sample aliquot. Measure the absorbance values of above aliquots against the reagents blank. Plot absorbance values against mg of silica per 100 ml of solution.

2.3.5 *Calculation* — Convert the photometric readings of the test solution to mg of SiO_2 by means of calibration curve and calculate the silica content in (500 ml) main solution *A* (**2.3.3**) as follows:

$$\begin{array}{l}\text{Total silica (as } SiO_2 \text{),}\\ \text{Percent by mass}\end{array} = \frac{(M_1 - M_2) + M_3}{M} \times 100$$

where

M_1 = mass in g of the crucible and the impure silica before hydrofluoric acid treatment,

M_2 = mass in g of the crucible and residue after hydrofluoric acid treatment,

M_3 = mass in g of residual silica present in 500 ml of the main sample solution (*A*), and

M = mass in g of the sample taken.

EXPLANATORY NOTE

Alumino silicate refractory materials contain alumina (Al_2O_3) and silica (SiO_2) in varying portions made synthetically by heating aluminium trifluoride at 1 000-1 200°C with silica and water vapour. •

It is used in kilns, laddles and furnaces that operate at higher temperature or under conditions for which fireclay refractories are not suitable.

This Indian Standard has been prepared in different parts to cover the chemical analysis of various constituents in alumino silicate refractory materials. The other parts of the standard are:

Part 1 Determination of loss on ignition
Part 3 Determination of aluminium
Part 4 Determination of phosphorus
Part 5 Determination of titanium
Part 6 Determination of iron
Part 7 Determination of manganese
Part 8 Determination of calcium and magnesium
Part 9 Determination of sodium and potassium

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